

## Data Validation Report

TDD No: 09-04-01-0011  
PAN: 001275.0440.01TA  
Site: El Dorado Hills  
Laboratory: Lab/Cor, Inc.  
Reviewer: Denise A. Shepperd, Trillium, Inc.  
Date: March 18, 2005

### I. Case Summary

#### SAMPLE INFORMATION:

Asbestos Samples: LC-AAMS-D08-100404; LC-AAMS-D09-100504; LC-AAMS-D10-100604; LC-AAMS-D11-100704; LC-AAMS-D12-100804; LC-SRA-2ZB-100804; LC-SRA-1ZB-100804; LC-SRA-FB-100804; LC-AAMS-D13-100904; LC-SRA-FB-101004; LC-AAMS-D14-101004; LC-NRA-2ZB-101104; LC-NRA-1ZB-101104; LC-AAMS-D15-101104; LC-AAMS-D16-101204; LC-NRA-1ZB-101204

Matrix: 16 Air samples

Analysis: Asbestos by Transmission Electron Microscopy

Collection Dates: October 4, 2004, through December 4, 2004

Sample Receipt Date: October 7, 2004

Analysis Date: December 1, 2004 through February 1, 2005

Analytical Method: ISO Method 10312

#### FIELD QC:

Field Trip Blanks (TB): LC-SRA-2ZB-100804; LC-SRA-1ZB-100804; LC-NRA-2ZB-101104; LC-NRA-1ZB-101104; and LC-NRA-1ZB-101204

Filter Blanks (FB): LC-SRA-FB-100804 and LC-NRA-FB-101004

Equipment Blanks (EB): None

Background Samples (BG): None

Field Duplicates (D1): Not Identified

#### TABLES:

1A: Analytical Results with Qualifications  
1B: Data Qualifier Definitions for Inorganic Data Review

#### SAMPLING ISSUES:

Three chain of custody (COC) documents were included in the data package. These documents included all of the field samples in the data package, as well as many additional samples. The chain of custody documents were properly completed with the exception that no indication was made of shipping method. The first person to sign off on the samples relinquished them on 10/14/04. The first signature for receipt was dated 10/15/04. Although it is probable that this person signed off on the samples when they were shipped to the laboratory, there is no indication on the COC as to the disposition of the samples between these two dates. As a result there is a break in the chain of custody for the samples in this data set.

## **VALIDATION PARAMETERS AND COMMENTS:**

### **I. Holding Times, Preservation and Sample Integrity**

This parameter is evaluated to ensure that sample custody is documented from collection through analysis, samples are analyzed within the recommended holding time, and that no alteration in sample content has occurred during sample shipment, handling, and storage.

There is no established holding time or storage condition for asbestos samples.

The integrity of the samples is dependent upon an unbroken chain of custody. Due to omissions on the COC records (discussed above) there is an apparent break in the chain of custody for the samples in this data set.

### **II. Calibration**

The analyses of materials of known content ensures that identification and quantitation of analytes will be accurate for all samples. Review of the documentation provided for appropriate calibration determines whether or not the analytical results reported by the laboratory are valid and supported by the data.

The data deliverables for this project were included in multiple data packages. No instrument calibration documentation was provided in association with the site sample data packages in this shipment. Documentation provided separately to support the identification and quantitation in the site samples in these data packages included the following:

A letter representing documentation of an NVLAP laboratory site assessment conducted on 11/7/03 was included in the data package. The letter included (dated 5/10/04) indicated that the laboratory met the on-site assessment requirements.

Results and evaluator notes and tables were included for an NISTIR 5351 analysis of an inter-laboratory QC sample. The laboratory's raw data were compiled and assessed by Batta Labs. Analysts were identified by initials and included two of the three analysts' initials documented with this sample set. "KM" and "DW" performed these PE sample analyses, "TM" and "JH" were not represented. According to the assessor's notes, the sample included chrysotile fibers and structures and the laboratory's results were within NVLAP and NISTIR 5351 acceptance limits. No raw data were provided for this QC sample.

Results for a New York State Department of Health Environmental Laboratory Approval Program proficiency test, conducted between 9/7/04 and 11/9/04, were also included. The proficiency samples included asbestos in air. The laboratory's results were satisfactory for all four of the air sample categories. Actinolite and amosite fiber types were identified and counts were acceptable according to the data sheet. No raw data were provided for this proficiency sample. Upon request, the laboratory provided raw data documenting the identification of actinolite and amosite asbestos on 1/27/05, in conjunction with the validation of a previous shipment of data packages. These data were inserted by the validator into the QC data package provided as supporting data with that previous shipment of data packages.

Documentation for a round-robin sample analyzed in the fall of 2004, by three separate laboratories, as part of the NVLAP requirements, was also included. The documentation included raw count sheets and reported results, as well as comparison with other laboratories' results. Results for all parameters were acceptable. The only analyst who participated in the analyses according to the documentation was "DW."

Instrument calibration information was not provided with the packages in this shipment. A previous shipment did include a separate calibration package which documented acceptable instrument calibration, including screen and

camera magnification, camera length and camera constant, spot size, k-factor, beam dose, EDS sensitivity and peak intensity. No documentation of grid opening size was provided. Documentation was provided in this separate proficiency and calibration data package for October through December, 2004, for both of the instruments used for analysis of samples included in this data package. As a result, analyses of the samples in this data set performed after that period are not supported by the calibration documentation provided.

Based on the fact that the laboratory demonstrated proficiency in the performance evaluation (PE) analyses performed in the third quarter of 2004, and that these PE samples included the two predominant asbestos types detected in this field sample set, no action was taken by the validator.

### **III. Blanks**

Sample matrices known to be devoid of the analytes of interest (method blanks) are prepared and analyzed with each analytical batch. Evaluation of this parameter ensures that contamination introduced during preparation and analyses is not attributed to the field samples.

Other blanks may be generated in the field or laboratory to ensure that no contamination is introduced during sampling and/or storage.

Blanks required for this project included Filter Blanks and Field Trip Blanks. Two Filter Blanks (LC-SRA-FB-100804 and LC-NRA-FB-101004) were included with this sample set. Five Field Trip Blanks (LC-SRA-2ZB-100804; LC-SRA-1ZB-100804; LC-NRA-2ZB-101104; LC-NRA-1ZB-101104; and LC-NRA-1ZB-101204) were also included. Field Trip Blanks and Filter Blanks are processed and analyzed by the laboratory in the same manner as field samples. Results can be used to assess contamination from a combination of the field and the laboratory environments. No asbestos structures were identified in any of the Filter or Field Trip Blanks submitted with this data set.

### **IV. Spiked Samples**

The analytes of interest are added in known concentrations to like-matrix blanks or authentic field samples before preparation. This parameter is evaluated in order to assess the laboratory's ability to preserve and recover the compounds of interest.

The analytical method does not require laboratory spiked sample analyses. It is recommended by the validator that some type of laboratory prepared or purchased spiked analyses be performed with each analytical sample batch.

The project requirements specified that results from the most recent inter-laboratory study would be acceptable as an LCS sample for these data. This requirement was met by the laboratory and reported results for the inter-laboratory study sample were acceptable for all air sample parameters (see Section I).

### **V. Duplicate/Replicate Samples**

Results for duplicate/replicate samples are evaluated to assess the laboratory's precision for the analytes of interest in the applicable sample matrix. For asbestos analyses, duplicate and replicate measurements take the form of a combination of variables which include the preparation of the grid, the choice of grid openings to be analyzed, and the analyst performing the counting and identification of structures.

The laboratory included all of the QC samples from all of the field sample sets in a separate data package under a separate report number (5906).

One of the two analysts, JH, not represented in the PE sample analyses included with the data packages for this project did perform intra-laboratory replicate and duplicate analyses on associated field samples. Results for these QC analyses for this analyst were within the sample-specific acceptance limits.

The quality assurance project plan (QAPP) requires five types of laboratory duplicate/replicate analyses, each to be performed at a rate of 5% (or one for every twenty) of the field samples. Based on 16 field samples reported in the data package, only one of each of these QC sample pairs were required. The laboratory compared the primary asbestos structure count for each of the QC samples prepared and analyzed. Results for all of the duplicate/replicate pair types were evaluated based on 95% confidence limits determined from the original sample count result. Results for all of the reported QC samples were within the laboratory's calculated limits. A summary of the laboratory QC samples included with this data set are as follows:

Replicate analyses:

- Two samples, LC-AAMS-D12-100804 and LC-AAMS-D15-101104, were analyzed as replicates, wherein a different preparation was analyzed by the same analyst;

Duplicate analyses:

- LC-AAMS-D15-101104 was analyzed as a duplicate, wherein the same grid openings were recounted by a different analyst;
- LC-AAMS-D12-100804 was analyzed as a duplicate, wherein different grid openings were selected for counting by a different analyst; and

No samples were analyzed as QC samples for two of the required categories:

- a replicate wherein different grid openings were selected by the same analyst for a second measurement
- a duplicate wherein a different analyst analyzes a different preparation.

One sample should have been included for each of these QC sample categories in order to satisfy the requirements of the QAPP.

An additional type of QC sample not identified by the QAPP was included. Both of the QC samples (LC-AAMS-D12-100804 and LC-AAMS-D15-100804) were recounted by the same analyst counting the same grids.

Comparison between results for two analyses by different analysts of the same grid openings (GO) from the same preparation of two of the samples showed variation in identification of asbestos types, morphology, and false positives and negatives for a number of the GOs counted. Despite these differences, however, the total asbestos counts showed excellent agreement.

The data user is cautioned that although the laboratory QC counts met the specified criteria, the acceptable range includes as much as a three-fold difference in asbestos concentrations for these samples. This range of variability is applicable to all asbestos results in the data set.

The count sheets for two analyses of LC-AAMS-D15-101104 which were listed as counts of the same grid openings, indicated that the same grid openings had not been counted in all cases. It is also possible that these represent transcription errors and not actual grid openings counted. From the documentation provided it is not possible to determine the nature of these errors, however, the total structure counts showed excellent agreement, varying by only one structure.

The QC summary form gives the original sample count for LC-AAMS-D12-100804 as 10, but the actual count is 8, according to both the raw data and the Form I for this sample. The validator corrected this result (from 10 to 8) and the acceptance range (from 4.80 - 18.39 to 3.45 - 15.8) on the QC summary form in the data package.

According to the QAPP provided with the data packages, field duplicates were required at a rate of 10% of field samples. Field duplicate pairs were not identified or evaluated as part of this validation effort.

## **VI. Identification**

Identification of asbestos structures and fibers is dependent on sample preparation techniques, analyst training, instrument operation, and data interpretation. Comparison with results from known standards is used to evaluate the accuracy of the structure identification for field samples.

Actinolite and chrysotile were identified in the field and QC samples. According to the report forms provided in the separate QC package, the laboratory correctly identified actinolite, chrysotile, and amosite in PE sample analyses performed in the third quarter of 2004. Comparison of identification between the various analysts, grid opening, and preparations combinations that make up the daily QC for these analyses were within acceptance limits. Therefore; based on the documentation provided, fiber and structure identifications for chrysotile and actinolite were determined to be valid as reported.

## **VII. Quantitation and Reported Detection Limits**

Raw data documentation is reviewed to ensure that all reported results and detection limits are correctly calculated, accurately reported, and supported by the raw data.

Results for asbestos categories, fiber density, and detection limits were correctly calculated and accurately reported by the laboratory. Results were verified by the validator using the information included on the reporting forms and the chain of custody records.

Three samples (LC-AAMS-D09-100504, LC-AAMS-D10-100604; and LC-AAMS-D16-101204) in this data set were rejected at analysis due to overloading of the filters with particulate material. According to the project work plan, these samples will be analyzed by the indirect method at a later time.

## **VIII. System Performance**

This parameter is evaluated to ensure that the laboratory analytical systems were functioning properly at the time of analyses and that methodology appropriate to the analyses were followed.

The analytical systems appear to have been working satisfactorily and to have been calibrated properly at the time of most these analyses, based on the documentation available from a previous shipment of data packages. Documentation was not available for instrument calibration for samples analyzed after 12/04.

## **IX. Documentation**

Data and documentation completeness is critical in providing support for the reported results. Problems encountered with the nature or quality of the data package documentation are addressed.

No raw data were provided in the data package for the proficiency samples analyzed in support of the laboratory's accreditation. Raw data to support the identification of actinolite and amosite were received upon request on 1/26/05 in conjunction with validation of a previous shipment of data from the same project.

Raw data for chrysotile fibers were not included in the data package for review. A separate package containing raw data for selected field samples from each laboratory lot was provided.

Count sheets included in the data package are computer generated forms. No date of the actual count is presented on these forms. If there is a corresponding bench sheet from which these forms are prepared, these should be supplied as a part of the data package. It is recommended that analyst's initials and date of count be added to the documentation.

The legend for the count sheets, which defines the codes used for the structure counts lists PSCH as the code for protocol chrysotile structures. The code appearing on the count sheets for this category is PCAS.

Raw data are an integral part of a complete and defensible data package. Edits made on all data should be performed correctly. Proper editing requires drawing a single line through the incorrect information, adding the correct information, and initialing and dating the changes.

Asbestos structures identified in the field and QC samples included actinolite and chrysotile. No examples of known materials were included in the data package in support of the sample analyses. Documentation provided separately included actinolite, chrysotile, and amosite, identified in the proficiency sample analyses. No raw data were provided for the proficiency sample analyses in the data package included in this shipment. Raw data were provided on 1/27/05 upon the request of the validator. These data were reviewed in conjunction with an earlier shipment of data packages for this project.

#### **COMMENTS:**

Based on the available data, results for all of the samples included in this data set were determined to be valid as reported by the laboratory. Reported results, analytical sensitivity, and detection limits are considered to be accurate within the bounds of the 95% confidence limits determined for each sample. No qualifiers were applied to these data by the validator.

#### **OTHER COMMENTS:**

Instrument calibration supplied in conjunction with an earlier shipment of data packages for this project only included analysis dates through 12/04. It is recommended that complete instrument calibration documentation be provided with every data package to fully support all of the sample results.

An unbroken chain of custody is critical to the integrity of all samples. It is recommended that when relinquishing samples for shipment, the sampler or sample custodian document this activity directly on the chain of custody in order to preserve the integrity of the samples. It is further recommended that the affiliations of all parties signing the records be identified to further support the samples' integrity.

It is recommended by the validator that performance data be included for each and every analyst involved in the analyses of samples for a data set. Due to both the limited information on calibration materials available and the limited requirements of the methods, the individual analysts' performance is the critical factor in the accuracy of the analyses.

Some of the required QC analyses were not performed for this individual data set. The validator considered all QC analyses for all of the data packages included in this shipment in the absence of complete QC data for the individual data packages.

The data results tables included as Table 1A include only the primary and total asbestos structure counts. Counts for individual categories required by the project Scope of Work are presented in the associated electronic data deliverables (EDD) tables.



This report was prepared according to the specifications of the analytical method, ISO Method 10312 “ Ambient air - Determination of asbestos fibres - Direct-transfer transmission electron microscopy method,” the document “USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review,” 2/94, and Trillium, Inc.’s SOP No. 0497-06A, for Validation of Analytical Data: Inorganic Analytes.

**TABLE 1B**

**DATA QUALIFIER DEFINITIONS FOR INORGANIC DATA REVIEW**

The definitions of the following qualifiers are prepared in accordance with the document, "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review," 2/94.

- U    The analyte was analyzed for, but was not detected above the level of the reported value. The reported value is either the sample quantitation limit or the sample detection limit.
- L    Indicates results which fall between the sample detection limit and the CRDL. Results are estimated and are considered qualitatively acceptable but quantitatively unreliable due to uncertainties in the analytical precision near the limit of detection.
- J    The associated value is an estimated quantity. The analyte was analyzed for and was positively identified, but the reported numerical value may not be consistent with the amount actually present in the environmental sample.
- R    The data are unusable. The analyte was analyzed for, but the presence or absence of the analyte cannot be verified.
- UJ   A combination of the "U" and "J" qualifier. The analyte was analyzed for but was not detected. The reported value is an estimate and may be inaccurate or imprecise.